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PRECOAT FILTRATION OF COAL LIQUID
FEASIBILITY STUDY OF BOTTOM ASH PRECOAT

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ABSTRACT

In this work, the suitability of using coal bottom ash as a precoat material for pressure filtration of liquefied coal was studied experimentally. Filtration of SRC I filter feed was accomplished in a laboratory filtration unit (1.5 in^2) using both coal bottom ashes and a commercial diatomaceous filter aid (celite 550). Filtration experiments were carried out at temperatures ranging from 150°C to 200°C, and pressure differentia ranging from 35 psid to 100 psid. Both materials were comparable in terms of the resistance of the cake and precoat media to filtration. Also both gave filtrates with less than 0.1% ash and 0.7% sulfur, thus meeting EPA combustion emission standards.

INTRODUCTION

For many coal liquefaction processes, precoat pressure filtration has been demonstrated as an effective technique for separating ash-forming materials as well as unconverted coal from the very viscous liquefied coal. Precoating the filter medium is necessary because the large amount of sub micron size particles could easily blind the pores of the filter medium. Currently, diatomaceous type filter aids are used as precoat materials. Although adequate supply of diatomite pose no major difficulty to the filter aid requirement for a major synthetic fuel facility, its relatively high price will render precoat pressure filtration in coal liquefaction unattractive. Based on the current price of diatomaceous filter aids, $160/ton, the annual costs of diatomaceous filter aid for a 25,000 TPD plant could range from $5 million to more than $75 million, depending on filtration process and specific equipment used in the process (1).

Innovative ideas such as use of raw coal, char (2,3) and fly ash (4,5) as economic substitutes of diatomaceous filter aid have been proposed. However, only the former are shown to be effective. The purpose of this study is to investigate experimentally the feasibility of using coal bottom ash as a precoat material for pressure filtration of liquefied coal. If coal bottom ash can be used effectively as a filter aid, the economics of precoat pressure filtration would be more attractive.

Filtration rate data, for both coal bottom ashes and a commercial precoat material, will be analyzed to determine the filtration parameters such as cake resistance and compressibility. These parameters, together with rate data and ash content of the filtrate, will be used as a basis to evaluate the relative performance of bottom ash as a precoat.

PRECOAT MATERIALS

A good filter aid, whether used as a body feed or as a precoat material should meet three basic requirements. First and the most important, it should be inexpensive. Secondly, it should have a narrow particle size range and a variety of particle shapes for achieving the desired cake permeability. Thirdly, it should be chemically and mechanically stable under operating process conditions and environments.

The coal bottom ash, composed primarily of coarser, heavier ash particles, is generally angular with a porous surface. While the chemical composition of coal bottom ash may vary due to geologic and geographic factors, the major constituents are silica, alumina, iron oxide and calcium oxide. For example, the Kentucky coals (Nos. 4, 9, and 11) produce bottom ashes that contain silica ranging from 46 to 52 percent and alumina ranging from 20 to 44 percent (6). Also, coal bottom ash will contain certain amounts of unburnt carbon or char. However, this should only enhance the bottom ash as a filter aid, since carbon itself is a very good filter aid.

EXPERIMENTAL

This experimental investigation was carried out in a laboratory bench-scale high temperature and pressure filtration apparatus (Figure 1). The filter had a 1.49 square inch effective filtration area. It was equipped with an in-line weighing device so that precise rate data could be obtained. In order to minimize filtrate vapor loss, the filter was also equipped with a pressurized filtrate receiver. The complete filter apparatus (Figure 2) was composed of two separate compartments. One compartment, equipped with a canopy exhaust hood, housed all of the

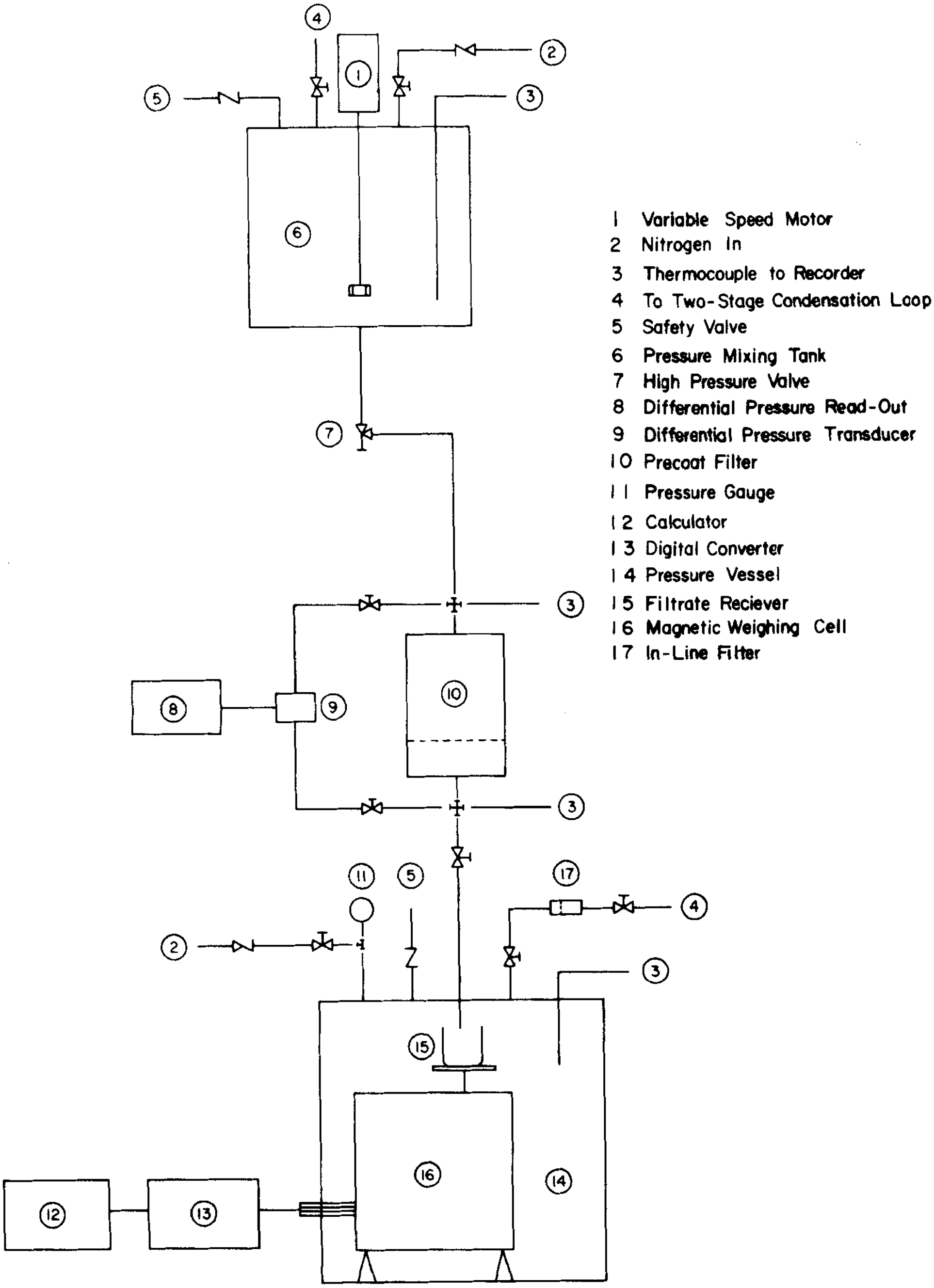

FIGURE 1. HIGH TEMPERATURE AND PRESSURE FLOW DIAGRAM

FIGURE 2. HIGH PRESSURE AND TEMPERATURE
PRECOAT FILTRATION EQUIPMENT

pressure vessels and interconnecting piping. The other compartment housed the process control and recording instruments.

The experimental operating procedure started with charging the slurry mixing tank with filter feed, heating up the system to the predetermined temperature, and pressurizing the feed mixing tank and filtrate receiving tank with nitrogen to the desired pressure levels. Then the filtration process was started. The filtrate collected in the receiver was weighed continuously by a load cell. A digital transducer converts signal to weight in grams which was recorded as a function of time. When the filtration was complete, a heated stream of nitrogen gas was introduced into the precoat filter until the load cell indicated no significant weight increase in the filtrate receiver. The filter was then cooled down by forced air circulation. The precoat filter was disassembled and contents removed for analysis. After cooling down to ambient temperature, the filtrate collected in the receiving tank was removed for ash and sulfur content analysis.

Precoating the filter support medium, which is a porous stainless steel, was done separately and prior to each experiment. The one inch precoat was applied by slurry deposition.

Bottom ashes generated from Kentucky coals were selected for this study. A commercial diatomaceous filter aid was also selected for a parallel study and served as a reference. In order to understand the particle size range effect on filtration, the ground bottom ash was classified into two different but narrower sizes. All three sizes of ash particle were used as precoat.

Experimental runs were done at various temperature and pressure differentials using SRC filter feed as feedstock. Filtration experiments carried out to date have been under maxium temperature of 200°C and maximum pressure differentia of 100 psi. Table 1 shows the experimental conditions and variables investigated for this feasibility study. The filter feed supplied by the Wilsonville SRC pilot plant was derived from Indiana V coal. It contains 4.30% ash and 1.04% sulfur.

DATA ANALYSIS

The data were treated in the following fashion, according to a well known equation of constant pressure filtration:

$$\frac{\theta}{(W/A)} = \frac{\mu\alpha\omega}{2\Delta p\rho}\left(\frac{W}{A}\right) + \frac{\mu R_m}{\Delta p\rho}$$

where

TABLE 1

Experimental Variables Outline

EXPERIMENT RUN	PRECOAT MATERIAL	PRECOAT SIZES: MESH	TEMP. °C	PRESSURE DIFFERENTIA PSI
1	None	N/A	150	50
2	Celite	$100/325^-$	150	50
3	Celite	$100/325^-$	200	100
4	Bottom Ash	$100/325^-$	150	35
5	Bottom Ash	$100/325^-$	150	50
6	Bottom Ash	$100/325^-$	175	50
7	Bottom Ash	$100/200^+$	150	100
8	Bottom Ash	$100/200^+$	200	50
9	Bottom Ash	$100/200^+$	200	100
10	Bottom Ash	$200/325^+$	150	100
11	Bottom Ash	$200/325^+$	200	100

μ = viscosity of filtration at filtration temperature

α = average specific cake resistance

ω = weight of solid per weight of filtrate

Δp = applied constant pressure differentia

Θ = filtration time

W = weight of filtrate collected

A = effective filtration area

Rm = filter medium resistance to filtrate flow

ρ = density of filtrate

A linear plot of the experimental quantities $\frac{\Theta}{W/A}$ versus W/A gave the following results:

$$\frac{\mu\alpha\omega}{2\Delta p\rho} = \text{slope of the line}$$

$$\frac{\mu Rm}{\Delta p\rho} = \text{ordinate intercept}$$

The least squares method was used to find the equation of the straight line (hence the slope and intercept). The specific cake resistance (α) and the filter medium resistance (Rm) were then calculated. Values of α and Rm for respective filter cakes and precoat media were then used as a basis for comparing coal bottom ash and the commercial filter aid.

RESULT AND DISCUSSION

Both the bottom ashes generated from Kentucky coal and a commercial diatomaceous filter aid were analyzed for their chemical and physical properties. The diatomaceous filter aid was Johns-Manville Celite 550. The analytical data are presented in Table

2. Bottom ash 1 was obtained from combustion of a 1:1 blend of an Eastern and Western Kentucky coal. Bottom ash 2 and 3 are two narrow mesh cuts obtained from classifying bottom ash 1.

In general, the coal bottom ashes had a grayish appearance due to a small amount of unburnt carbon. The difference in chemical

TABLE 2

Properties of Kentucky Coal Bottom Ashes and Diatomite Filter Aid

	BOTTOM ASH[1]			DIATOMITE[2]
ANALYSIS	BA1	BA2	BA3	CELITE 550
PHYSICAL				
% Moisture	0.1	0.19	0.25	0.35
% Ash	95.8	96.1	96.6	99.45
% Volatile Matter (Ignition Loss)	0.9	1.0	1.2	(0.2)
% Fixed Carbon	3.2	2.7	2.8	0
Density (g/ml)	1.17	1.15	1.16	0.45
Color	Gray	Gray	Gray	White
CHEMICAL				
% SiO_2	53.9	60.4	59.4	89.6
% Al_2O_3	28.2	29.6	28.4	3.96
% Fe_2O_3	7.4	7.3	7.05	1.37
% TiO_2	4.14	1.34	2.32	0.17
% P_2O_5	0.15	0.29	0.28	0.30
% CaO	1.16	1.19	1.20	0.63
% MgO	0.65	0.64	0.69	0.73
% (Na_2O + K_2O)	2.23	1.26	2.43	3.23
SCREEN SIZE, MESH				
% > 100	0	0	0	5.98
% 100/150	0.3	1.3	0	3.27
% 150/200	23.2	98.7	0	9.16
% 200/325	22.3	0	100	52.79
% < 325	54.2	0	0	28.80

1. Ash from combustion of 1:1 blend of Eastern and Western Kentucky Coal.
2. Johns-Manville Celite 550.

composition between diatomaceous and coal ashes is the high alumina, titanium oxide and iron oxide content in coal ashes. By far the most significant difference is the particle shape and surface characteristics. Scanning electron microscopic pictures showed that the Celite 550 particles exhibited a variety of shapes and highly porous surface characteristics. Coal bottom ashes exhibited fewer variety of shapes and had a more refined porous surface structure.

The filtration rate data are presented in the fashion described previously for different process conditions in Figures 3 to 7. Filter precoat made from coal bottom ash, in all three particle size ranges, were able to yield initial filtration rate comparable to those yielded by using diatomite precoat.

Initial filtration rate data are presented in Table 3. Initial filtration rates were calculated from that portion of the lines closest to the ordinate, yielding the highest rates. Initial filtration data are more applicable to rotary drum precoat filtration where a layer of blinded precoat and cake are continuously removed. For leaf precoat filtration, because of rapidly decreasing filtration rate due to both precoat pore blinding and increasing cake resistance, the entire section of filtration line must be considered.

The quality of separation was evaluated by considering the ash and sulfur content of the filtrate. These data are presented in

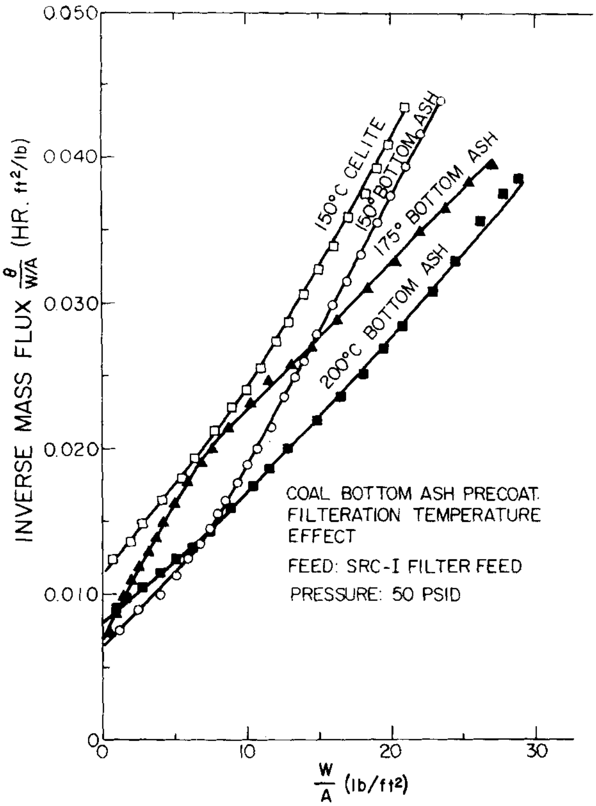

FIGURE 3: TEMPERATURE EFFECT ON SRC-I COAL SLURRY FILTRATION.

Table 4. Both coal bottom ash and Celite precoat were able to reduce ash to much less than 0.1 wt.% and sulfur less than 0.7 wt.%. One experiment was carried out without use of any precoat, resulting in an increase in ash content to 0.26%. However, filtration runs with precoat made of 200/325 mesh coal bottom ash produced filtrate containing unacceptable high ash content. This result strongly suggests that extremely fine and narrow size range ash particles are not a good filter aid.

TABLE 3

Initial Filtration Rate

EXPERIMENT RUN	PRECOAT	FILTRATION RATE LB/HR/FT^2
1	None	270
2	Celite-550	90
3	Celite-550	235
4	Ash 100/325$^-$	154
5	Ash 100/325$^-$	156
6	Ash 100/325$^-$	141
7	Ash 100/200$^+$	346
8	Ash 100/200$^+$	119
9	Ash 100/200$^+$	209
10	Ash 200/325$^+$	206
11	Ash 200/325$^+$	160

TABLE 4

Filtrate Quality and SRC Recovery
Feed: SRC-1 Filter Feed Contains 4.30% Ash and 1.04% Sulfur

EXPERIMENT		FILTRATE ANALYSIS		SRC RECOVERY**
RUN	PRECOAT	ASH WT.%*	SUFLUR WT.%	WT.%
1	None	0.26	0.55	89
2	Celite-550	0.06	0.62	--
3	Celite-550	0.05	0.60	90
4	Ash 100/325$^-$	0.02	0.47	--
5	Ash 100/325$^-$	0.03	0.54	--
6	Ash 100/325$^-$	0.02	0.62	89
7	Ash 100/200$^+$	0.05	0.50	85
8	Ash 100/200$^+$	0.05	0.63	--
9	Ash 100/200$^+$	0.04	0.60	84
10	Ash 200/325$^+$	0.26	0.51	85
11	Ash 200/325$^+$	0.22	0.52	87

* Analytical data indicated error limits of 0.01 to 0.02%.

** SRC Recovery =

$$\frac{\text{Total filtrate weight collected}}{\text{Feed wt. - (Total cake wt. - total THF soluable in cake)}}$$

SRC product recovery was calculated when possible. Generally, a better than 85% recovery is achieved. Viscosities of filtrate measured at 100°C, 150°C and 200°C are 18.9, 6.2 and 2.8 centipoise, respectively.

The temperature effect upon coal bottom ash precoat filtration can be seen from Figure 3. In general, the higher temperature reduces viscosity and should increase the filtration rate. Other factors, such as α, could offset the temperature effect over the temperature range tested. The initial filtration rate is not greatly influenced, but secondary filtration rate (beyond the slope change point) follows that general trend. Higher temperature (> 200°C) experiments will be carried out in order to better assess the temperature effect.

The effect of pressure differential on filtration is shown in Figures 4 and 5. At the temperature levels (150°C and 200°C), larger pressure differentials provide a greater driving force and hence higher filtration rates.

Three different particle sizes range coal bottom ashes (Table 2) were used to make precoats. As indicated in Table 4, all ash precoats except those made of 200/325 particle size are effective in separating ash and sulfur containing solids. Particle size effect on precoat's performance in filtering liquefied coal is shown in Figures 6 and 7. At operating conditions, precoats made of narrower size range ash particles give higher filtration rates. Particularly the precoat made of less than 100 mesh but larger

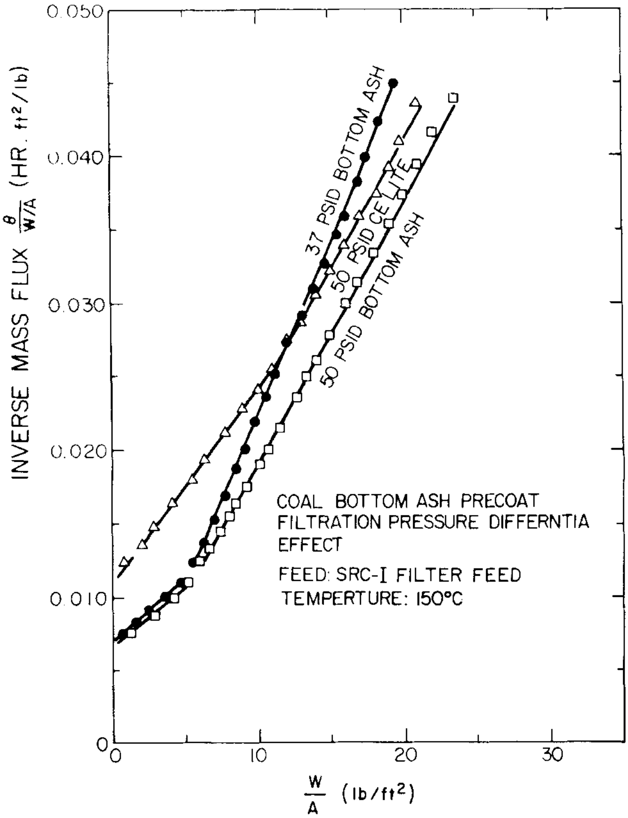

FIGURE 4: PRESSURE DIFFERENTIA EFFECT ON SRC-I COAL SLURRY FILTERATION

than 200 mesh size ash particles gives a much higher filtration rate.

A close examination of the filtration rate lines presented in Figure 3 through Figure 7 reveal that most of lines are not straight and the data can be better represented by two straight lines. This phenomena was also experienced by other investigators

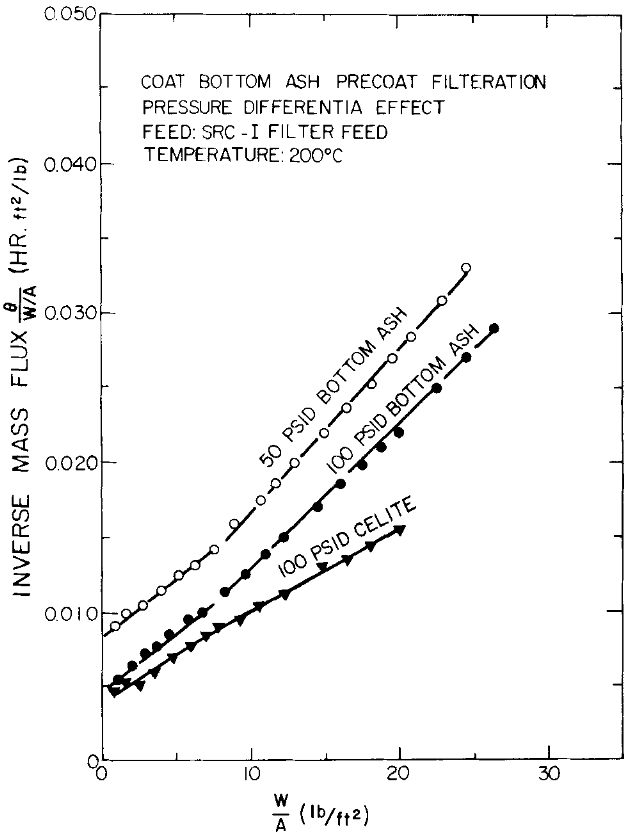

FIGURE 5: PRESSURE DIFFERENTIA EFFECT ON SRC-I COAL SLURRY FILTERATURE

(1) who concluded that the specific cake resistance is responsible for the change of slope. The definition of α is

$$\alpha = \frac{K_1 (1-\varepsilon) So^2}{\rho_p \varepsilon^3}$$

where K_1 = constant and equals 4.167 for random particles of definite size and shape

ε = porosity of the bed

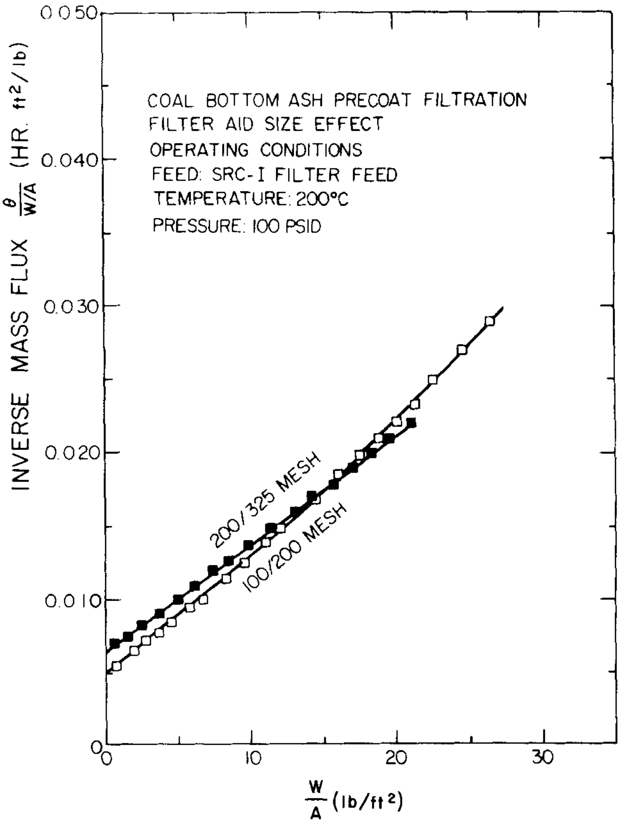

FIGURE 6: COAL BOTTOM ASH PRECOAT PARTICLE SIZE EFFECT OF FILTRATION

So = specific area of the solid particle per volume of particle

ρ_p = density of solid particle in the cake

During a specific experiment, the "collapse" of cake bed may occur and thus alter porosity of the cake bed. The cake compressibility may be further examined via the following relationship

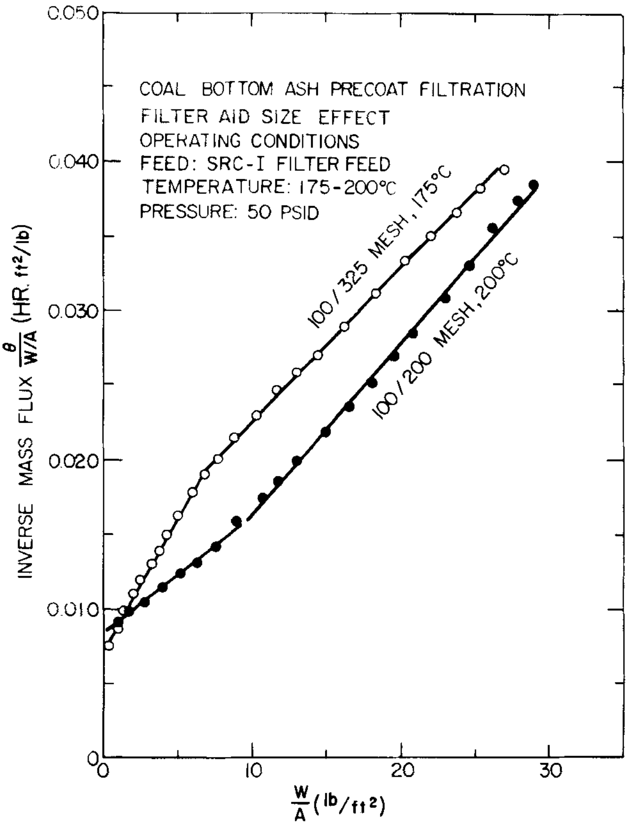

FIGURE 7-COAL BOTTOM ASH PRECOAT PARTICLE SIZE EFFECT ON FILTRATION

$$\alpha = \alpha' (\Delta P)^{\gamma}$$

where α' = a constant dependent on particle size

γ = cake compresibility

The above equation may be rewritten in the following form;

$$\ln \alpha = \ln \alpha' + \gamma \ln \Delta P$$

Cake compressibility values may be obtained from the slope if one plots $\ln \alpha$ vs. $\ln \Delta P$. Such a diagram is shown in Figure 8.

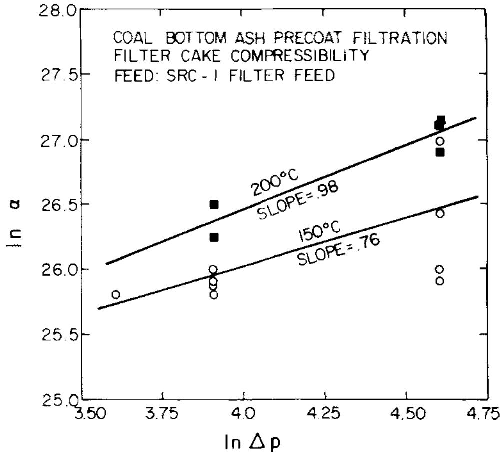

FIGURE 8: FILTER CAKE COMPRESSIBILITY OF SRC-I SLURRY

It is seen that cake compressibility at 200°C is 0.98, and 0.76 at 150°C. This indicated a highly compressible cake is formed during the filtration of SRC-1 liquefied coal. This explains why filtration rate in all cases studied drop rapidly as the cake starts to build up.

Table 5 shows the specific cake resistance and medium (precoat plus s.s. support plate) resistance calculated from slope and intercept according to the filtration equation. These values are calculated using only the first section of the filtration line. Medium resistance data indicates that precoat made of coal bottom ashes offers no more resistance than do those precoats made of diatomite.

TABLE 5

Precoat Filtration Parameter

EXPERIMENT RUN	SPECIFIC CAKE RESISTANCE $\alpha \times 10^{-11}$ ft/lbm	MEDIUM RESISTANCE $R_m \times 10^{-10}$ 1/ft
1	3.56	4.43
2	2.13	13.9
3	4.83	23.5
4	1.59	6.0
5	1.59	8.02
6	3.69	13.8
7	2.98	7.22
8	2.49	23.2
9	6.17	26.4
10	7.23	12.1
11	5.87	34.5

SUMMARY

Coal bottom ashes, simply crushed to about 100 mesh size, can be used as an economical filter aid in a coal liquefaction solid separation process. Experimental results show that precoat pressure filtration using bottom ash precoat yield filtrate containing ash less than 0.1 wt.% and sulfur less than 0.7 wt.%. These low ash and sulfur contents will meet the Environmental Protection Agency's combustion emission standards. However, precoat made of extremely fine and narrow size range ash particles shows an inability to give an effective separation. Filtration rates generated from ash precoat filtration are comparable to those rates obtained using diatomite precoat. Precoat made of ash particles larger than 200 mesh but smaller than 100 mesh were able

to improve the filtration rates while maintaining excellent solid separation efficiency. Also, results show that the ash precoat medium resistance is approximately the same as that of diatomite precoat.

This study has demonstrated that coal bottom ash is an excellent substitute for traditional diatomaceous filter aid. By utilizing coal bottom ash as a filter aid, not only is precoat filtration process economy significantly improved, but also there will be no foreign material to contaminate the filtration products. Filter cake may be used as a supplimental solid fuel and ash may be recovered for recycling.

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